भारतीय मानक

Indian Standard

IS 495 : 2023

स्नेहक के लिए ग्रेफाईट फ्लेक — विशिष्टि

(दूसरा पुनरीक्षण)

Graphite Flake for Lubricants — Specification

(Second Revision)

ICS 75.100

भारतीय मानक ब्यूरो
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November 2023 **Price Group 6**

Lubricants and Their Related Products Sectional Committee, PCD 25

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Lubricants and Their Related Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1954 and subsequently revised in 1967. In the first revision, method of test for loss of heating and non-graphite carbons and sampling were modified. Considerable assistance has been derived from Indian defence service specification, IM 1690(b) Graphite; flake; issued by Ministry of Defence and material specification number DTD 806 : 1950 Graphited grease issued by the Ministry of Aviation, UK.

In this second revision, requirement for volatile matter and fineness have been included in Table 1. Further, to improve the values of neutrality of graphite flakes, the requirement of acidity of water extract, water soluble matter, petroleum ether-soluble matter, and ash content have been modified. In view of the changes in requirements, methods of tests have also been suitably modified. The fineness by sieving test has been included in the table instead of separate requirement and has been modified due to non-availability of graphite of lesser pore size.

For determining fineness, the aperture sizes of sieves are based on IS 460 (Part 1) : 2020 'Test sieves — Specification: Part 1 Wire cloth test sieves (*fourth revision*)'. Where these sieves are not available, other equivalent standard sieves, as judged by the aperture may be used.

This standard contains **4.1**, which calls for agreement between the purchaser and the supplier.

The composition of the Committee responsible for the formulation of this standard is given in Annex J.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

GRAPHITE FLAKE FOR LUBRICANTS — SPECIFICATION

(Second Revision)

1 SCOPE

This Indian Standard prescribes the requirements and the methods of sampling and test for graphite flake, intended for use in the manufacture of lubricants.

2 REFERENCES

The standards given below contain provisions, which through reference in this text, constitute the provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards:

IS No.	*Title*
IS 323 : 2009	Rectified spirit for industrial use — Specification (*second revision*)
IS 460 (Part 1) : 2020	Test sieves — Specification: Part 1 Wire cloth test sieves (*fourth revision*)
IS 1447 (Part 1) : 2021	Methods of sampling of petroleum and its products: Part 1 Manual sampling (*second revision*)
IS 1745 : 2018	Petroleum hydrocarbon solvent — Specification (*third revision*)
IS 3025 (Part 11) : 2022	Methods of sampling and test (physical and chemical) for water and waste water: Part 11 *p*H value (*second revision*)

3 REQUIREMENTS

3.1 General

The material shall be dry, free flowing, steel grey in colour, having a metallic lusture, and a smooth and soapy feel. It shall be free from grit, foreign matter, grease, oil and visible impurities.

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in col (4) of the Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable containers as agreed to between the purchaser and the supplier.

4.2 Marking

4.2.1 The containers shall be marked with the following information:

a) Name of the material;
b) Manufacturer's name, initials or trade-mark, if any;
c) Net mass of material;
d) Identification in code or otherwise to enable the lot of consignment or manufacture to be traced back from records; and
e) Any other statutory requirements.

4.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

Representative samples of the materials shall be drawn as prescribed in IS 1447 (Part 1).

Table 1 Requirement for Graphite Flake for Lubricants

(*Foreword and Clause* 3.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Annex/IS
(1)	(2)	(3)	(4)
i)	Volatile matter (2 h at 105 °C ± 2 °C), percent by mass, *Max*	1.25	A
ii)	Petroleum ether-soluble matter, percent by mass, *Max*	0.25	B
iii)	Water-soluble matter, percent by mass, *Max*	0.25	C
iv)	Reaction with water extract		
	a) Alkalinity to phenolphthalein, *Max*	0.02	D
	b) Acidity to methyl orange, *Max*	0.02	
	or		
	*p*H of water extract	5.5 to 8.5	IS 3025 (Part 11)
v)	Ash, percent by mass, *Max*	1.0	E
vi)	Non-graphitic carbon, percent by mass, *Max*	3.0	F
vii)	Freedom from abrasive matter	To pass the test	G
viii)	Fineness, by sieving test:		
	Retained on a 150 micron sieve, percent by mass, *Max*	Nil	H
	Retained on a 75 micron sieve, percent by mass, *Max*	2	
	or		
	Average particle size (D50), *Max*	10	Particle size analyzer

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF VOLATILE MATTER

A-1 PROCEDURE

A-1.1 Weigh accurately 5 g (to nearest of 0.000 1 g) of the sample (W_1) into a glass dish (approximately 50 mm diameter and 30 mm in height) fitted with a ground glass lid and then reweigh glass dish (W_2)

A-1.2 Transfer the dish to an oven maintained at 105 °C ± 2 °C and remove the lid. Allow the sample to remain in the oven for 2 h.

A-1.3 At the end of 2 h remove the dish from the oven, replace the stopper, and cool in a desiccator. Momentarily release the stopper on the weighing dish to ensure that the pressure inside is the same as atmospheric and then reweigh (W_3).

A-2 CALCULATION

Volatile matter, percent by mass =

$$\frac{W_2 - W_3}{W_1} \times 100$$

ANNEX B

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF PETROLEUM ETHER SOLUBLE MATTER

B-1 REAGENT

B-1.1 Petroleum Ether — conforming to grade 60/80 of IS 1745.

B-2 PROCEDURE

Weigh accurately 5 g (to nearest of 0.000 1 g) of the sample (W_4) and extract with petroleum ether in a soxhlet apparatus for 2 h. Weigh an empty flask (W_5) and filter the petroleum ether extract through a filter paper into a flask. Distil off the solvent and remove the last trace of it in vacuum. Cool the flask and weigh to constant weight (W_6).

B-3 CALCULATION

Petroleum ether-soluble matter, percent by mass =

$$\frac{W_6 - W_5}{W_4} \times 100$$

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF WATER SOLUBLE MATTER

C-1 PROCEDURE

Transfer 20.0 g ± 0.1 g of the sample (W_7) to a 250 ml stoppered measuring cylinder. Add 200 ml of water and shake the cylinder for 1 h. Allow the solid matter to settle, and decant the supernatant liquid through a No. 42 Whatman filter paper, rejecting the first 10 ml. Using a pipette withdraw 50 ml of the filtrate and run it into a pre-weighted glass beaker (W_8). Evaporate the solution to dryness on a boiling water bath, transfer the basin to an oven maintained at 105 °C ± 2 °C for 3 h. Remove the basin from the oven and allow to cool in a desiccator containing freshly activated silica gel and reweigh (W_9). Reserve the remainder of the filtrate for the determination of reaction of water extract (*see* Annex D).

C-2 CALCULATION

Water soluble matter, percent by =

$$\frac{W_9 - W_8}{W_7} \times 100$$

ANNEX D

[*Table* 1, *Sl No*. (iv)]

DETERMINATION OF REACTION OF WATER EXTRACT

D-1 REAGENT

D-1.1 Hydrochloric Acid (HCl) — 0.1 N

D-1.2 Sodium Hydroxide Solution — 0.1 N

D-1.3 Phenolphthalein Indicator

Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit (*see* IS 323) and dilute with water to 100 ml.

D-1.4 Methyl Orange Indicator — 0.04 g/100 ml of rectified sprit (*see* IS 323)

D-2 PROCEDURE

Use the water reserved from Annex C. Using a pipette withdraw two 25 ml aliquot portions of the solution and transfer to separate 250 ml conical flasks. To the first flask add a few drops of the phenolphthalein solution and, if the reaction is alkaline, titrate the solution with the hydrochloric acid solution (V_1), volume used in ml. Carry out a blank determination without the Graphite (V_2), volume used in ml. If the solution is acidic to phenolphthalein, add a few drops of methyl orange indicator to the second aliquot portion and titrate the solution with the sodium hydroxide solution (V_3), volume used in ml. Carry out a blank determination without the Graphite (V_4), volume used in ml.

D-3 CALCULATION

Alkalinity, percent (m/v, as sodium hydroxide) =

$$= \frac{(V_1 - V_2) \times N_1 \times E_1}{25 \times 10}$$

Acidity, percent (m/v, as sulphuric acid) =

$$\frac{(V_3 - V_4) \times N_2 \times E_2}{25 \times 10}$$

where

N_1 = normality of the hydrochloric acid solution;

E_1 = equivalent weight of sodium hydroxide;

N_2 = normality of the sodium hydroxide solution;

E_2 = equivalent weight of sulphuric acid.

ANNEX E

[*Table* 1, *Sl No*. (v)]

DETERMINATION OF ASH

E-1 PROCEDURE

Weigh accurately about 2 g ± 0.05 g (to nearest of 0.0001 g) of the material (W_{10}) in a pre-weighed crucible (W_{11}). Ignite in a muffle furnace at 900 °C to 950 °C until all the graphite has been burnt off. Cool the crucible in a desiccator and reweigh. Repeat the ignition, cooling and weighing until the mass is constant (W_{12}).

E-2 CALCULATION

Ash, percent by mass = $\frac{W_{12} - W_{11}}{W_{10}} \times 100$

ANNEX F

[*Table* 1, *Sl No*. (vi)]

DETERMINATION OF NON-GRAPHITE CARBON

F-1 APPARATUS

F-1.1 Muffle Furnace — having a thermal regulator and maintained at 440 °C ± 5 °C.

F-2 PROCEDURE

Weigh accurately about 2 g (to nearest of 0.0001 g) of the material (W_{13}) into a silica dish fitted with cover and note the weight of the dish with the material (W_{14}). Place the dish in the centre of the furnace in such a manner that the dish occupies 40 percent of the centre area of the muffle furnace. Heat the sample for 16 h in the furnace, then cool it and its contents in a desiccator and weigh.

NOTE — The heating for 16 h should preferably be done at a stretch. If this is not practicable, it should be done in two equal installments of 8 h each on two successive working days.

F-3 CALCULATION

Non-graphite carbon, percent by mass =

$$\left(\frac{W_{14} - W_{15}}{W_{13}} \times 100\right) - (A + B)$$

where

W_{14} = first weight mass, in g, of the dish and sample;

W_{15} = second mass, in g, of the dish and sample;

W_{13} = mass of sample;

A = percent loss on heating [volatile matter (*see* Annex A)]; and

B = percent petroleum ether-soluble matter (*see* Annex B)

ANNEX G

[*Table* 1, *Sl No*. (vii)]

TEST FOR FREEDOM FROM ABRASIVE MATTER

G-1 PROCEDURE

G-1.1 Take a pair of flat circular brass plates with chamfered edges and of approximately 75 mm diameter, polished successively with fine emery cloth and finally with rouge to give a mirror surface. Weigh about 2 g of the material, squeeze it between the polished faces of the brass plates and rub then vigorously for 1 min. Wash the plates with a suitable solvent and examine for visible scratching.

G-1.2 The material shall be taken to have satisfied the requirements of the test, if there are no visible scratches on any of the two plates.

ANNEX H

[*Table* 1, *Sl No*. (viii)]

METHOD FOR SIEVING TEST

H-1 APPARATUS

H-1.1 Sieves 150-micron — [*see* IS 460 (Part 1)]

H-1.2 Sieves 75-micron — [*see* IS 460 (Part 1)]

H-1.3 Varnish brush — 25 mm

H-2 PROCEDURE

Place 10 g of the sample (W_{16}) on the appropriate sieve and brush gently with the varnish brush for 15 min or until no more sample passes the sieve, whichever is the shorter period. Weigh any material retained on the sieve (W_{17}).

H-3 CALCULATION

Material retained on the sieve (150 or 75 micron), percent by mass =

$$\frac{W_{17}}{W_{16}} \times 100$$

ANNEX J

(*Foreword*)

COMMITTEE COMPOSITION

Lubricants and Their Related Products Sectional Committee, PCD 25

Organization	*Representative(s)*
In Personal Capacity (*Flat - 1002, Raheja Heights, D - Wing, off Gen A K Vaidya Marg, Dindoshi, Malad East Mumbai - 400097*)	DR Y. P. RAO **(*Chairperson*)**
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Member Secretary
SHRIMATI KREETI DAS
SCIENTIST 'C'/DEPUTY DIRECTOR
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Amendments Issued Since Publication

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